

Effects of DBDS and DBPC antioxidants on the corrosion of copper strips immersed in transformer oil

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ABSTRACT

This paper presents the experimental findings on the effects of antioxidants on the corrosion of copper strips immersed in mineral oil (MO)–antioxidant samples. First, the uninhibited MO was mixed thoroughly with dibenzyl disulphide (DBDS) or 2,6-di-tert-butyl-p-cresol (DBPC) at different concentrations (5, 15, 25, 50, and 200 mg/kg) using a hot plate magnetic stirrer set at a temperature of 73 °C and stirring speed of 750 rpm for 15 min. Following this, the MO–antioxidant samples were poured into separate test vessels and copper strips were added into the vessels. Next, each MO–antioxidant sample was blanketed with nitrogen gas, sealed, and placed in a forced convection laboratory oven. The MO–antioxidant samples were then thermally aged at 150 °C for 48 h. The results showed that the acidity of the MO–antioxidant sample decreased with an increase in the antioxidant concentration, regardless whether the antioxidant was DBDS or DBPC. However, the corrosion of the copper strip worsened with an increase in the antioxidant concentration, where DBDS had a higher relative degree of corrosivity to copper compared with DBPC. In addition, the results showed that a DBPC concentration of 25 mg/kg reduced the acidity of the MO–antioxidant sample and resulted in a moderate tarnish of the copper strip.

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1. INTRODUCTION

During the operation of oil-immersed transformers, the transformer mineral oil (MO) tends to degrade over time due to heating, moisture, and oxidation, resulting in poor dielectric properties and interfacial tension. In addition, heat, electrical, and chemical stresses increase the acidity of the MO and produce sludge. The degradation process of MO can be reduced by adding performance-enhancing additives. The general amount of additive used to decelerate the oxidation process is 0.3% [1]–[3]. Examples of additives are; i) antioxidants such as dibenzyl disulphide (DBDS), 2,6-di-tert-butyl-p-cresol (DBPC), and 2,6-di-tert-butylphenol (DBP); ii) metal passivators such as 1,2,3-benzotriazole (BTA) and Irgamet® 39 [1]–[3]; and iii) pour point depressants such as Lubrizol® 7671A, Viscoplex® 10-171, and Viscoplex® 10-310 [4]. Antioxidants retard the oxidation process while metal passivators prevent the formation of copper sulphide on the surface of the copper components in the transformer. Pour point depressants reduce the pour point of the MO. Even though antioxidants help decelerate the oxidation process, studies have shown that

DBDS causes corrosion on metals (copper windings and silver components). DBDS is known to be a corrosive sulphur compound, along with elemental sulphur, mercaptans, sulphides, and thiophenes. The reactivity of sulphur compounds varies from the most reactive sulphur to the most stable sulphur according to the following order: elemental sulphur>mercaptans>sulphides>disulphides>thiophenes [5]–[8].

Studies have been carried out to mitigate sulphur corrosion due to sulphur-containing MOs. For example, Ito and Morishima [1] studied the extension of corrosion control time due to the synergistic effect of BTA (30 ppm) and DBPC (0.3%) on the insulating oil containing 300 ppm of DBDS. Liu *et al.* [9] studied the effect of introducing DBPC into MO containing DBDS and the MO sample was then subjected to accelerated thermal ageing tests. They found that deposition of copper sulphide on the paper insulation significantly increased for DBPC concentrations less than 0.87%. However, the deposition of copper sulphide on the paper insulation decreased with a further increase in the DBPC concentration. Mehanna *et al.* [2] assessed the effect of introducing various antioxidants (DBPC, DBP, DBDS, 2-t-BPC, N-phenyl-1-naphthylamine, BTA, and methylated BTA) into the MO. Oxidation stability tests were carried out according to the ASTM D2440 standard [10] and the results showed that DBPC was the most suitable antioxidant for MO, followed by DBP. Jaber *et al.* [3] studied the effects of combining various antioxidants (DBDS, DBPC, BTA, and DBP) in the MO. Copper strips wrapped with kraft paper were immersed in the MO samples and aged at 150 °C for 5 days. The concentrations of antioxidants were fixed as follows: i) DBDS (150 mg/L), ii) BTA (30 mg/L), iii) DBPC (0.3%), and iv) DBP (0.3%).

Even though studies [2], [3] have been conducted on the effects of adding antioxidants in MO on the corrosion of copper strips, the antioxidant concentrations were fixed and little is known on the effects of adding antioxidants (particularly DBDS and DBPC) at different concentrations on the corrosion of copper strips immersed in the MO–antioxidant samples. Therefore, this study is aimed to address this gap, where DBDS and DBPC antioxidants were added into uninhibited MO at different concentrations (5, 15, 25, 50, and 200 mg/kg). After the MO–antioxidant samples were prepared, copper strips were added into the samples and then aged at 150 °C for 48 h in accordance with the ASTM D1275 standard (Method B) [11]. Following this, the effects of each antioxidant on the acidity and corrosion of the copper strips were determined.

2. MATERIALS AND METHOD

The materials, measurements of the moisture content, acidity, and AC breakdown voltage of the MO are presented in this section. The preparation of the MO–antioxidant samples, and tests used to assess the presence of corrosive sulphur in the MO–antioxidant samples are also described. The presence of corrosive sulphur was assessed in terms of the acidity of the MO–antioxidant samples and corrosion of the copper strips immersed in the MO–antioxidant samples.

2.1. Materials

The MO used in this study was Nytro Libra uninhibited MO (Nynas AB, Sweden) [12]. The antioxidants used were i) DBDS and ii) DBPC. The chemical structures of DBDS and DBPC are shown in Figures 1(a) and (b). The melting point of DBDS is 69–72 °C while the melting point of DBPC is 70 °C [13]. Before the MO and antioxidants were mixed, the MO was treated so that its moisture content was ≤ 35 mg/kg, which is the permissible moisture content limit for MOs [14]–[16]. This was achieved by blanketing the MO with nitrogen gas [17] until the moisture content of the MO was acceptable. In addition, the acidity and AC breakdown voltage of the MO were measured to ensure that the acidity was ≤ 0.03 mg KOH/g (permissible limit for acidity) and the AC breakdown voltage was ≥ 20 kV (permissible limit for AC breakdown voltage for an electrode gap distance of 1 mm). These limits are specified in the ASTM D3487 standard [18].

2.2. Measurements of the moisture content, acidity, and AC breakdown voltage of the MO

The moisture content of the MO was measured according to the ASTM D1533 standard [19] using Metrohm 899 coulometer for coulometric Karl Fischer titrations. The acidity of the MO was measured according to the ASTM D974 standard [20] using Metrohm 848 Titrino plus compact titrator. The moisture and acidity measurements were conducted three times for the MO and the mean values were determined. The AC breakdown voltage of the MO was determined using Megger OTS60PB portable oil tester in accordance with the ASTM D1816 standard [21]. Unlike moisture content and acidity, the mean AC breakdown voltage is the average of five individual measurements in sequence according to the ASTM D1816 standard.

2.3. Preparation of the MO–antioxidant samples

The MO was mixed with the antioxidants using a hot plate magnetic stirrer for 15 min, where the temperature and stirring speed of the magnetic stirrer were set at 73 °C and 750 rpm, respectively. The

concentrations of the DBDS and DBPC antioxidants (in mg) for all samples are tabulated in Table 1. The volume of each MO sample was 500 mL, which was equivalent to 440 g since the density of the MO was 0.88 g/cm³. After the mixing process, each MO–antioxidant sample was poured into an amber bottle, blanketed with nitrogen gas, and stored in a designated area.

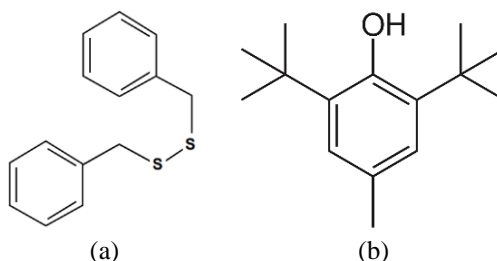


Figure 1. Chemical structures of; (a) DBDS [5], [22] and (b) DBPC [23]

Table 1. Concentrations of DBDS and DBPC antioxidants for all samples

Sample	Antioxidant	Antioxidant concentration	
		(mg/kg)	(mg)
1	—	0	0.0
2	DBDS	5	2.2
3		15	6.6
4		25	11.0
5		50	22.0
6		200	88.0
7	DBPC	5	2.2
8		15	6.6
9		25	11.0
10		50	22.0
11		200	88.0

2.4. Accelerated thermal ageing test

For the accelerated thermal ageing test, 250 mL of MO–antioxidant sample was poured into a test vessel. Following this, a copper strip (length × width × thickness: 6 mm × 25 mm × 0.5 mm) was immersed in the MO–antioxidant sample. In this study, the copper strips were prepared according to the following procedure. First, the copper strips were polished using silicon carbide sandpapers with a grit size of 240. Next, each copper strip was bent into a V shape with an angle of ~60°. The V-shaped copper strips were then washed with acetone and distilled water. Finally, the V-shaped copper strips were dried in a forced convection laboratory oven at 100 °C for 5 min. Once the copper strips were immersed in the MO–antioxidant samples, the oil surface was blanketed with nitrogen gas. The test vessels were then sealed with polytetrafluoroethylene caps equipped with fluoroelastomer O-rings.

Following this, the test vessels containing the MO–antioxidant samples and copper strips were placed in a forced convection laboratory oven and aged at 150 °C for 48 h. The accelerated thermal ageing test was performed according to the ASTM D1275 standard (Method B) (Standard Test Method for Corrosive Sulfur in Electrical Insulating Liquids). After the accelerated thermal ageing test, the test vessels were taken out from the laboratory oven and left to cool down to room temperature. Once the MO–antioxidant samples had cooled, the V-shaped copper strips were taken out from the test vessels, washed with acetone, and dried. The copper strips were then classified by comparing their colour against the ASTM copper strip corrosion standard, in accordance with the ASTM D130 standard [24].

3. RESULTS AND DISCUSSION

In this study, after the MO was treated, the moisture content, acidity, and AC breakdown voltage of the MO were measured. Following this, the treated MO was mixed with DBDS or DBDC antioxidant at different concentrations, resulting in 11 MO–antioxidant samples (Table 1). It shall be noted that DBDS or DBPC was not added into Sample 1, and this sample served as a control sample. A copper strip was then added into each MO–antioxidant sample, and the samples were aged at 150 °C for 48 h. Following this, the acidity of the aged MO–antioxidant samples was measured while the copper strips were classified according to the ASTM D130 standard [24].

3.1. Moisture content, acidity, and AC breakdown voltage of the treated MO

The properties of the MO after moisture treatment are presented in Table 2. The moisture, acidity, and AC breakdown voltage of the MO were found to be 25.9 mg/kg, 0.0268 mg KOH/g, and 22 kV respectively, which complied with the permissible limits prescribed in the ASTM D3487 standard: i) moisture content ≤ 35 mg/kg, ii) acidity ≤ 0.03 mg KOH/g, and iii) AC breakdown voltage ≥ 20 kV for an electrode gap distance of 1 mm.

Table 2. Properties of the treated MO

Property	Unit	MO
Moisture	mg/kg	25.9
Acidity	mg KOH/g	0.0268
AC breakdown voltage	kV	22

3.2. Effect of DBDS and DBPC antioxidants on the acidity of the MO

The MO–antioxidant samples along with the V-shaped copper strips were aged at 150 °C for 48 h. After the accelerated thermal ageing test, the acidity of the MO–antioxidant samples were measured and the results are shown in Figure 2. It can be observed that the acidity of the aged MO added with either DBDS or DBPC showed a similar trend. The change in acidity can be divided into three distinct regions: i) the acidity increased up to a maximum value when the antioxidant concentration was 5 mg/kg, ii) the acidity then decreased when the antioxidant concentration was within a range of 5–25 mg/kg, and iii) the acidity showed a minor decrease (where the trend was relatively invariant) when the antioxidant concentration was more than 50 mg/kg. When the antioxidant concentrations were 5 and 15 mg/kg, the acidity of the aged MO–antioxidant samples containing DBDS was higher compared with that for samples containing DBPC. In contrast, the acidity of the aged MO–antioxidant samples containing DBPC was higher than that of samples containing DBDS for an antioxidant concentration of 25, 50, and 200 mg/kg. It can be deduced that DBPC results in lower acidity at low antioxidant concentrations (≤ 25 mg/kg) whereas DBDS results in lower acidity at higher antioxidant concentrations (≥ 25 mg/kg).

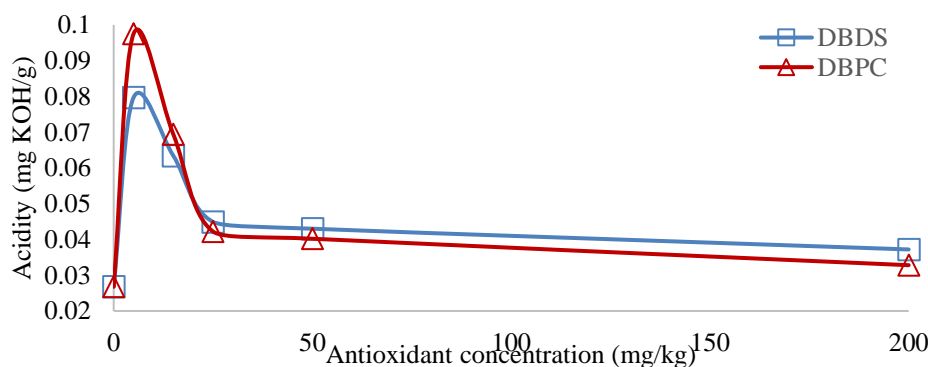


Figure 2. Acidity of the aged MO–antioxidant samples

3.3. Effect of DBDS and DBPC antioxidants on the corrosion of copper strips

After accelerated thermal ageing test, the copper strips were classified according to the ASTM copper strip corrosion standard and the results are summarized in Table 3. In general, the results showed the effect of DBDS and DBPC antioxidants on the corrosion of copper strips immersed in the MO–antioxidant samples. It can be seen that copper strips immersed in Sample 1 (MO), Sample 2 (MO + 5 mg/kg of DBDS), Sample 7 (MO + 5 mg/kg of DBPC), Sample 8 (MO + 15 mg/kg of DBPC), and Sample 9 (MO + 25 mg/kg of DBPC) had moderate tarnish, where the copper strips were multi-coloured with the presence of lavender blue or silver or both, overlaid on claret red. The copper strips immersed in Sample 3 (MO + 15 mg/kg of DBDS), Sample 4 (MO + 25 mg/kg of DBDS), Sample 5 (MO + 50 mg/kg of DBDS), Sample 10 (MO + 50 mg/kg of DBPC), and Sample 11 (MO + 200 mg/kg of DBPC) had dark tarnish, where the copper strips were multi-coloured with the presence of red and green (reminiscent of a peacock), but without grey. Only the copper strip immersed in Sample 6 (MO + 200 mg/kg of DBDS) was corroded, as indicated by its graphite black or lustreless black appearance. Based on the results, it can be deduced that the addition of DBDS (up to 5 mg/kg) and DBPC (up to

25 mg/kg) into the MO do not play a role in the corrosion of copper strips since the copper strips are moderately tarnished similar to the appearance of the copper strip immersed in Sample 1(MO). However, increasing the DBDS or DBDC beyond these concentrations influence the corrosion of copper.

Table 3. Classification of the copper strips after accelerated thermal ageing test

Sample	Composition	Classification of copper strip
1	MO	Moderate tarnish (2c)
2	MO + 5 mg/kg of DBDS	Moderate tarnish (2c)
3	MO + 15 mg/kg of DBDS	Dark tarnish (3b)
4	MO + 25 mg/kg of DBDS	Dark tarnish (3b)
5	MO + 50 mg/kg of DBDS	Dark tarnish (3b)
6	MO + 200 mg/kg of DBDS	Corrosion (4b)
7	MO + 5 mg/kg of DBPC	Moderate tarnish (2c)
8	MO + 15 mg/kg of DBPC	Moderate tarnish (2c)
9	MO + 25 mg/kg of DBPC	Moderate tarnish (2c)
10	MO + 50 mg/kg of DBPC	Dark tarnish (3a)
11	MO + 200 mg/kg of DBPC	Dark tarnish (3b)

3.4. Relationship between acidity of the MO–antioxidant samples and corrosion of copper strips immersed in the MO–antioxidant samples

Figure 3 shows the relationship between the acidity of aged MO added with antioxidant (either DBDS or DBPC) at different concentrations and the corresponding classification of copper strip corrosion. It shall be highlighted that the acidity of the MO should be ≤ 0.20 mg KOH/g [25] because beyond this level, the rate of the oil–paper insulation will increase rapidly. In this study, the sample with the highest acidity (0.0975 mg KOH/g) was Sample 7 (MO + 5 mg/kg of DBPC). The acidity of MO sample containing DBPC decreased with an increase in the DBPC concentration, as indicated by the following sequence: Sample 7>Sample 8>Sample 9>Sample 10>Sample 11. Likewise, the MO samples containing DBDS showed the same trend as that for the DBPC containing samples, where the acidity was highest (0.0797 mg KOH/g) for Sample 2 (MO + 5 mg/kg of DBDS) and the acidity decreased with an increase in the DBDS concentration according to the following sequence: Sample 2>Sample 3>Sample 4>Sample 5>Sample 6.

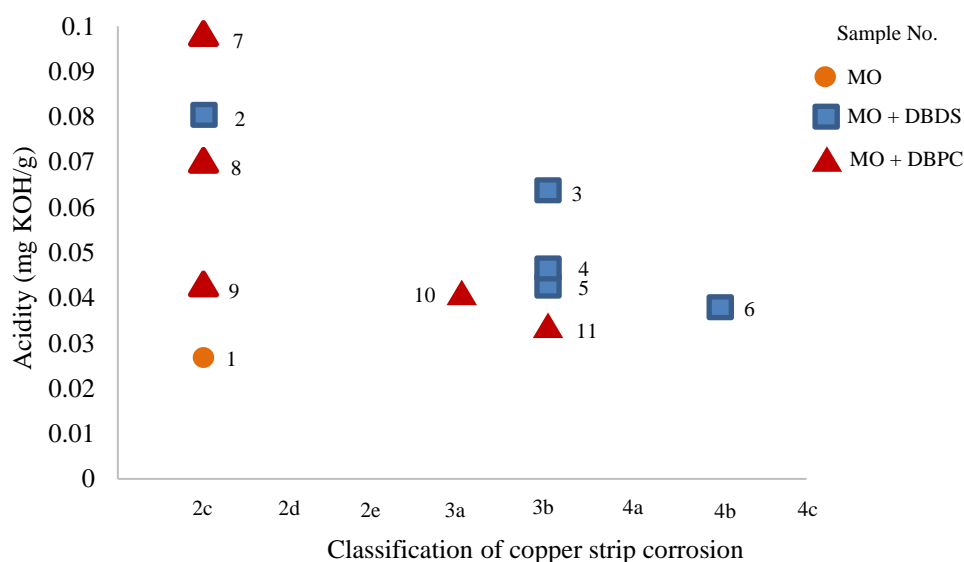


Figure 3. Acidity of the aged MO–antioxidant samples and their corresponding classification of copper strip corrosion. Note: that the numeric value next to each data marker denotes the sample number

According to the ASTM copper strip corrosion standard, the condition of the copper strip is categorized into four main categories: i) slight tarnish, ii) moderate tarnish, iii) dark tarnish, and iv) corrosion. In this study, it was found that the corrosion of the copper strip worsened as the antioxidant concentration was increased, regardless whether the antioxidant was DBDS or DBPC. The addition of DBDS

into the MO resulted in moderate tarnish (Level 2c) and dark tarnish (Level 3b) and the highest DBDS concentration (200 mg/kg) resulted in corrosion (Level 4b). In contrast, the addition of DBPC into the MO only resulted in a dark tarnish (Level 3b) for the highest DBPC concentration (200 mg/kg). Hence, it is evident that the use of DBDS as an antioxidant in MO has a more pronounced effect on the corrosion of the copper strips. Furthermore, the addition of DBPC into MO at a concentration of 25 mg/kg (i.e., Sample 9) gives the best results, where the acidity of the aged sample is the lowest (0.0422 mg KOH/g) and the copper strip immersed in this sample is moderately tarnished. In contrast, DBDS is a more aggressive type of sulphur compound compared with DBPC, which may be attributed to its intrinsic properties as a sulphide antioxidant whereas DBPC is a phenolic antioxidant.

4. CONCLUSION

In this study, the effects of DBDS and DBPC antioxidants on the corrosion of copper strips were investigated. MO-antioxidant samples were prepared by mixing treated MO with antioxidants (DBDS or DBPC) at different concentrations (5, 15, 25, 50, and 200 mg/kg). Following this, copper strips were prepared and immersed in the MO-antioxidant samples. The MO-antioxidant samples were then subjected to accelerated thermal ageing test at 150 °C for 48 h. After the accelerated thermal ageing test, the acidity of aged MO-antioxidant samples were measured and the relative degree of corrositivity of the MO-antioxidant samples to the copper strips was classified. Based on the experimental results, it is concluded that increasing the antioxidant concentration reduces the acidity of the MO, but this comes at the expense of a higher degree of corrositivity on the copper strip. The best antioxidant that reduces the acidity of the MO and results in a moderately tarnished copper strip is DBPC with a concentration of 25 mg/kg.

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